BROWNING

Tests of some Illinois Fire Clays

Ceramic Engineering

B. S.

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TESTS OF SOME ILLINOIS FIRE CLAYS

BY

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THESIS

FOR THE

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THIS IS TO CERTIFY THAT THE THESIS PREPARED UNDER MY SUPERVISION BY

Thomas Samuel Browning.

ENTITLED Tests of Some Illinois Fire Clays.

IS APPROVED BY ME AS FULFILLING THIS PART OF THE REQUIREMENTS FOR THE

DEGREE OF Bachelor of Science

in Ceramic Engineering.

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PROPERTIES OF REFRACTORY CLAYS.

Refractory clays are of two distinct types, the plastic and the non-plastic. The former are used as a bonding material in the manufacture of refractory products, while the non-plastic is incorporated to increase the porosity and reduce the shrinkage.

The plastic clays include the kaolins or china clays, the ball clays and fire-clay. Kaolins are white burning clays approaching the mineral kaolinite in chemical composition. They vitrify only at high temperatures, and have a very high softening point, while these have excellent refractory qualities; they are not sufficiently plastic and have not been used to any great extent in refractory products.

The ball clays are exceedingly plastic and make excellent bond clays. They are used more or less in such wares as glass pots, crucibles etc. They may be classed as dense burning clays, since they attain a low porosity at low temperatures but they have a fairly high softening temperature. They have in some cases been used to excellent advantage in the manufacture of fire brick.

The term fire-clay primarily refers to clays capable of withstanding a high temperature, but this term has come to be used to distinguish clays which are usually found underlying coal seams and are considered to represent the soil in which the coal producing vegetation grew. Under this very poor definition

we may distinguish the fire-clays according to their refractory qualities as number one, two or three. (In some instances, a class four has been introduced.)

Purdy[1] places fire-clays in these classes according to their porosity,-temperature relation and burning, as shown by the curves in Fig. I. While Bleininger[2] and Brown in a later investigation have added to this a classification based on the viscosity of the clay when burned under load conditions.

Bleininger and Brown's specifications based on results of load tests."--From the results of the work done in this laboratory the following tentative specifications are suggested. In this connection the No. 1 clay refractories are divided into two classes, A and B. The first includes those materials for which both refractoriness and load-carrying ability is required; the second those where refractoriness is demanded but compressive strength at furnace temperatures is not a main requisite. The No. 2 clays are supposed to include those products which are somewhat inferior to the high-grade refractories, but which nevertheless form an important class of products suitable for many uses.

No. 1 A.--Materials of this class should show, when tested in the ordinary manner in the Deville or an electric furnace and heated at a rate so that the final temperature is obtained in not less than one hour, a softening temperature of not less than

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1 Ill. State Geological Survey Bul. 9, p. 270.

2 Tech. Paper No. 7, Bureau of Standards, p. 76.

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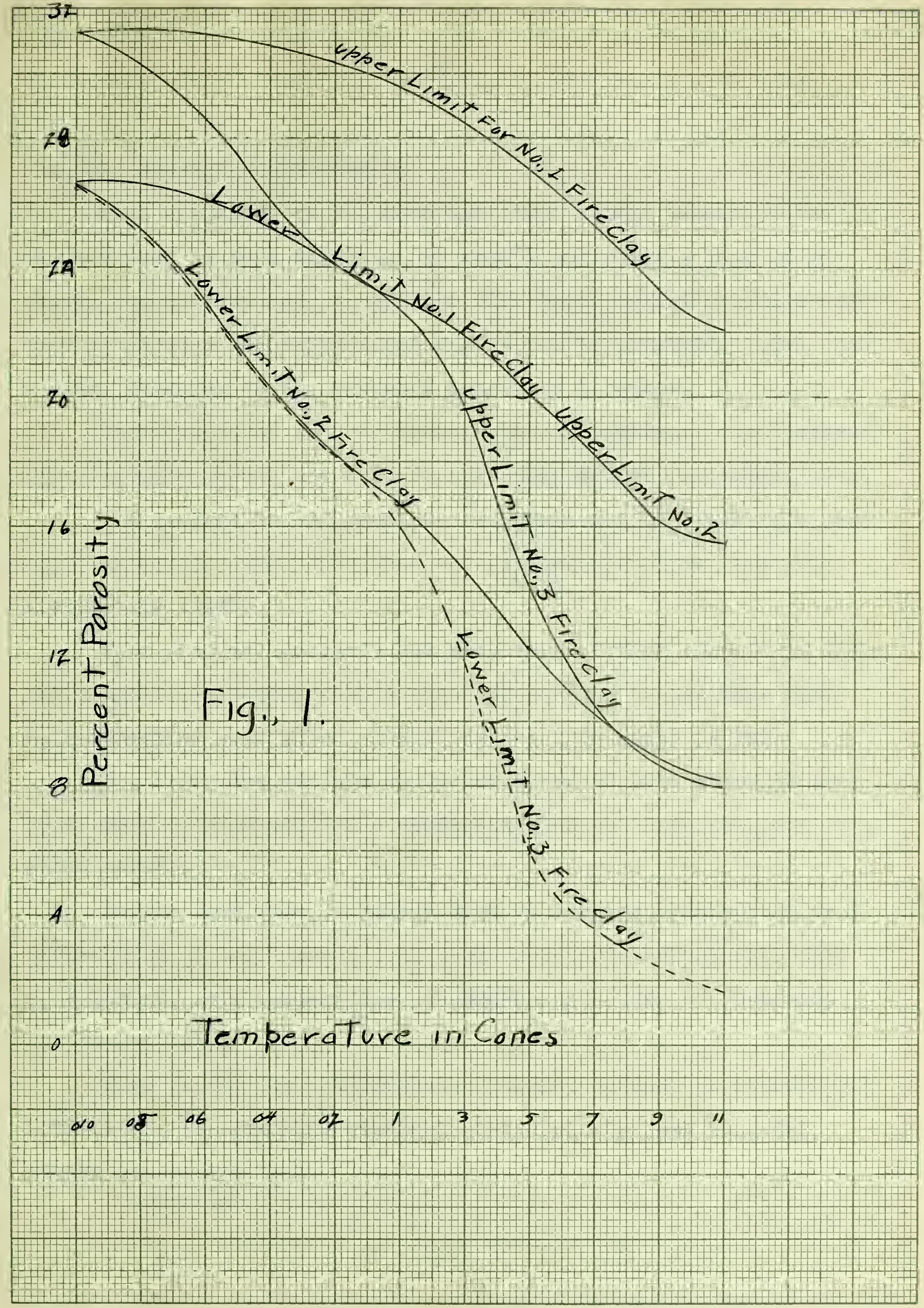

Upper Limit For No. 1 Fire Clay
Lower Limit No. 1 Fire clay
Upper Limit No. 2
Lower Limit No. 2 Fire Clay
Upper Limit No. 3 Fire clay
Lower Limit No. 3 Fire clay
Percent Porosity
Temperature in Cones
32
28
24
20
16
12
8
4
0
010
08
06
04
02
1
3
5
7
9
11

Fig., 1.

cone 31, approximately 1690°C.

When subjected to the load test in a manner substantially as described in this bulletin, at 1350°C and under a load of 50 pounds per square inch, a standard fire brick tested on end should show no serious deformation and should not be compressed more than 1 inch, referred to the standard length of 9 inches.

When tested on end, at atmospheric temperature, the compressive strength should not be less than 1000 pounds per square inch.

The product in its manufacture should not be fired to a maximum temperature lower than that corresponding to cone 11, or approximately 1350°C.

Upon chemical analysis the empirical formula calculated from the composition should show a total RO content of not more than 0.22 molecular equivalent, including the iron oxide as FeO.

No. 1 B.--The average softening point of this class of products should correspond to a temperature of not less than cone 31, about 1690°C.

In the load test it should show no serious deformation or a contraction of more than 1 inch, referred to the standard length of 9 inches, at a temperature of 1350°C and a load of 30 pounds per square inch.

When tested for compressive strength, on end, at atmospheric temperature the crushing strength should not be less than 800 pounds per square inch.

The product should not have been fired to a maximum temperature lower than that represented by cone 10, about 1300°C.

The chemical formula calculated from the analysis should show a content of RO fluxes of not more than 0.22 equivalent.

One important point has been left unconsidered in these specifications, namely, the question of shrinkage or expansion upon heating fire bricks to higher temperatures. It was thought that the data at hand were insufficient to suggest any requirements.

No. 2. Refractories.--The softening point of this class of refractories should not be lower than the temperature corresponding to cone 28, approximately 1630°C.

In the load test the materials of this classification should be able to carry a load of 25 pounds per square inch at 1300°C without serious deformation or a contraction greater than 1 inch, referred to the standard length of 9 inches.

The chemical formula, as calculated from the analysis, should not show a content of more than 0.32 equivalent of fluxes.

It is seen from these requirements that the limits drawn are closer than those generally considered for No. 2 fire-clays. However it is believed that with these restrictions this class of refractories would become more generally useful in industrial application."

The non-plastic refractory clays are represented by the flint and semi-flint fire-clays. These are hard and flint-like

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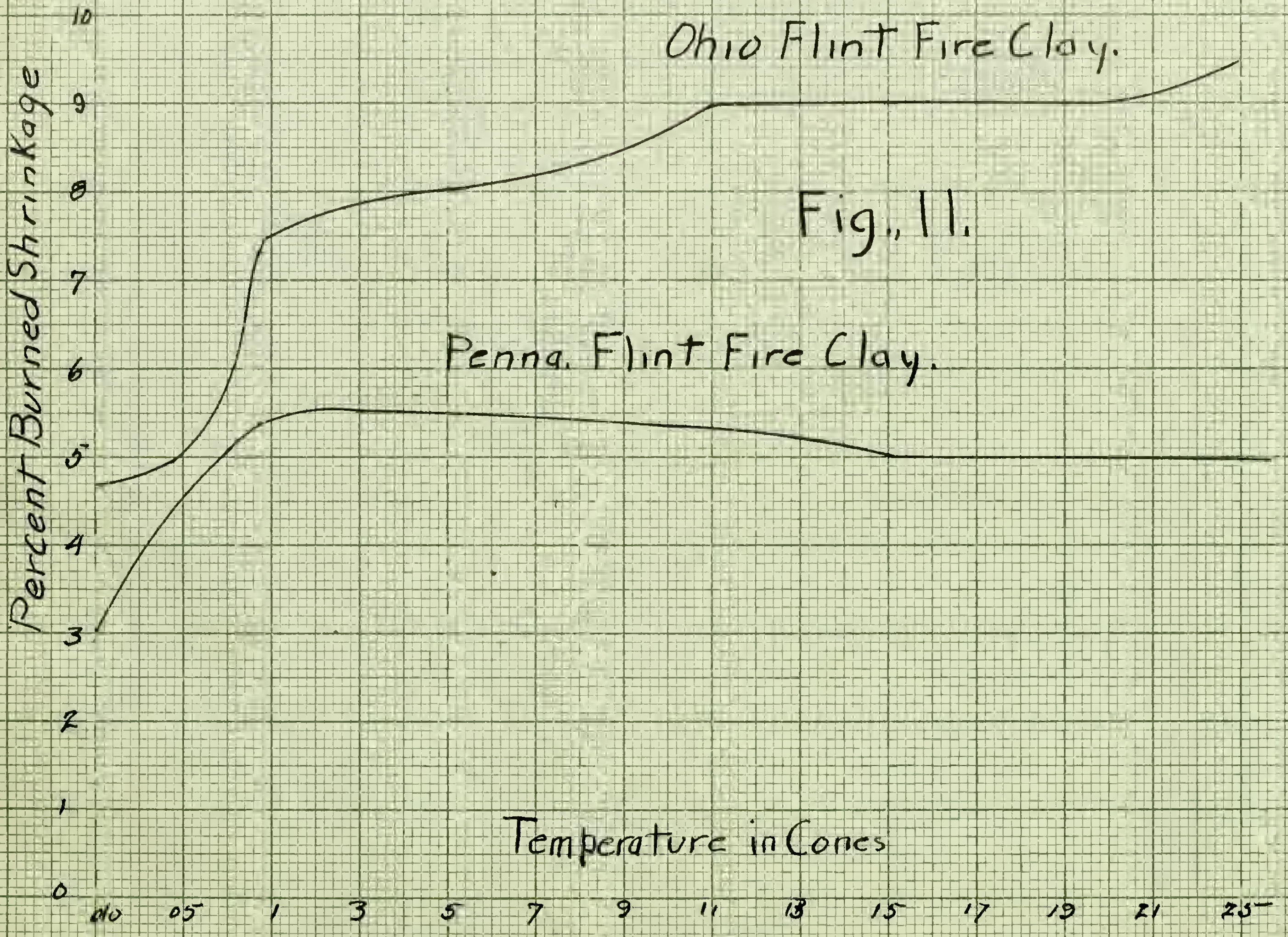
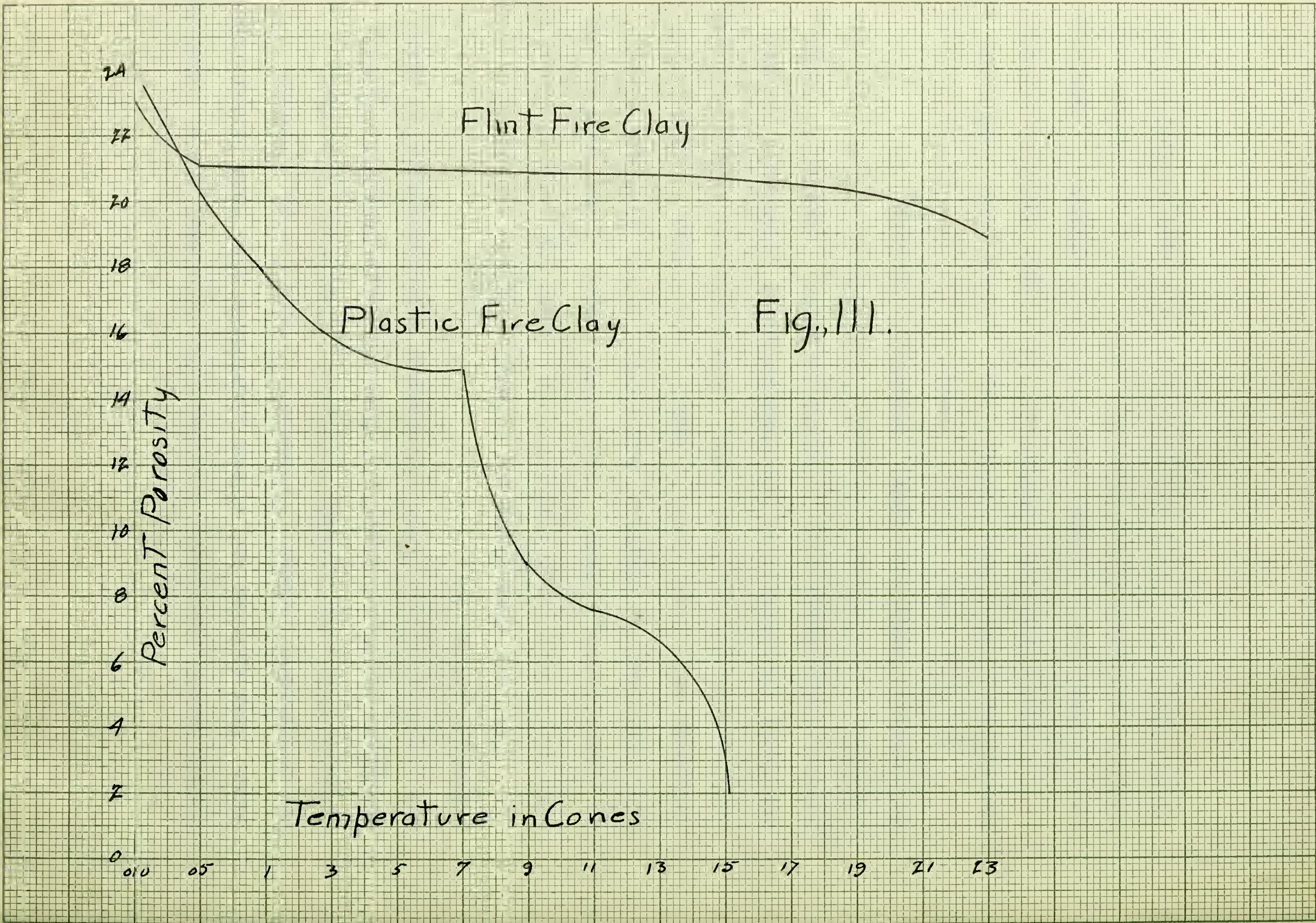


Percent Burned Shrinkage
0
1
2
3
4
5
6
7
8
9
10
Temperature in Cones
010
05
1
3
5
7
9
11
13
15
17
19
21
25
Ohio Flint Fire Clay.
Penna. Flint Fire Clay.

Fig., II.



Flint Fire Clay
Plastic Fire Clay
Percent Porosity
Temperature in Cones
24
22
20
18
16
14
12
10
8
6
4
2
0
010
05
1
3
5
7
9
11
13
15
17
19
21
23

Fig., III.

with a smooth conchoidal fracture like flint and one of dense texture. They develop but little plasticity even when finely ground, but are usually highly refractory and have a high porosity, and a comparatively high burning shrinkage. Characteristic fire shrinkage curves of two flint fire-clays are shown in Fig. II; the relation of the flint and plastic types according to their porosity in Fig. III.

Illinois fire-clays are generally of the plastic type. Many beds have been found throughout the state and some of these are at present being worked. The general scope of the beds can be best described by starting near Morris on the Illinois River, following this river down to LaSalle and thence to Rock Island and south to St. Louis or below. The clays are thought to follow generally the outcrop of the Pennsylvanian formations and are usually found where coal is known to exist. Some of the most important districts are centered around Ottawa, Utica, Oglesby, Colchester, Roodhouse, Drake, Whitehall and Anna.

The clay mined in Union County in the Anna district is of the nature of ball clay and has found considerable commercial use in the manufacture of retorts and pots. That found around Whitehall is used quite extensively in the manufacture of stoneware, While clays from many of the other districts are used for terra cotta, pots etc. The clays which are used most extensively, as

bond clays for fire brick and refractory shapes are found in the districts around Ottawa and Oglesby.

Object of Tests.

In order to learn more about Illinois fire-clay resources and as a means of determining the qualities of some of the fire-clays of different sections of the state, the following tests have been made:

1. Slaking qualities,
2. Plasticity,
3. Drying behavior,
4. Burning shrinkage,
5. Porosity,
6. Softening point,
7. Behavior under load at high temperatures.

Tests for spalling, thermal conductivity, slagging and resistance to changes of temperature as given by Bell and Wright[3] were not made since they apply more directly to the comparison of different brick than to the testing of bond clays.

Qualifications of Refractory Bond Clays.

1. Fair slaking ability,
2. Good plasticity,
3. Fair drying shrinkage) no warping
4. " burning ")
5. (a) Fairly high porosity when burned to (cone 11 or higher),
   (b) Low for pot clays,
6. High softening point (cone 28 as minimum and better, cone 30 or higher)
7. Should conform to Bleininger and Brown's specifications for load capacity given on page 2.

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3 Brick & Clay Record.

To be of commercial value, a fire-clay should conform to these qualifications. The slaking ability and plasticity must be good, otherwise the dried piece will be hard to mold and will not hold its shape when dry. The burning and drying shrinkages must be low in order that the finished pieces will not be cracked, warped or strained. For certain purposes such as glass pot manufacture, the clay must be free from contaminating materials, such as iron, lime and manganese. The softening point must of course be high, otherwise the primary value of the clay is destroyed. Porosity must also be high, for all bond clays used for bricks and similar products, but for glass pots and crucibles it should be low.

The load test is a measurement of viscosity. This should be high, or the clay will be deformed when subjected to high temperatures.

Description of Clays Studied.

Of the three clays studied, two are coal measure clays and one is separated from a coal vein by a thin bed of limestone. Samples Nos. 1 and 2 are from the same locality, near Oglesby in LaSalle County. No. 1 underlies a forty inch vein of number 2 coal and rests upon a thin bed of limestone, under which lies clay No. 2. Clay No. 3 is a coal measure clay from Ottawa, and is at present mined and used by a company making refractories.

Description of Samples as Received.

-1-

1. Color--dark gray-blue,
2. Hardness--soft and brittle,
3. Slaking behavior--slakes with ease,
4. Plasticity--good,
5. Carbonaceous matter--large amount,
6. Other impurities--contains a large amount of pyrites in lumps varying from specks up to 2 and 3 inches in diameter, nearly all of which can be eliminated by blunging the clay with water and passing through an 80 mesh screen.

-2-

1. Color--dark gray,
2. Slaking behavior--slakes easily,
3. Hardness--harder than No. 1; brittle and has flinty appearance,
4. Plasticity--good,
5. Carbonaceous matter--little,
6. Other impurities--none obvious in rough examination.

-3-

1. Color--gray,
2. Hardness--harder than No. 1 or 2, and has flinty appearance,
3. Slaking behavior--slakes easily,
4. Plasticity--good,
5. Carbonaceous matter--little,
6. Other impurities--none obvious in rough examination.

Description of Tests.

Plasticity and ease of working up of the clays were merely estimated when the test pieces were made, since all the manufacturer requires in this respect is the comparative ability of a clay to work up with water to a plastic mass and retain its shape

when dry enough; tensile strength in this condition being necessary of course to allow the piece to be handled without danger of breaking.

Drying behavior--Each clay was ground between rolls to pass a 20 mesh screen and pugged by hand with just sufficient water to make a plastic mass of about the consistency of stiff mud. This was expressed through a die, 1" x 1" by means of a hand power plunger press. The column was cut to form briquettes 4" long by means of a wire. Shrinkage marks were made 8 cm. apart on the wet pieces, which were then placed in a steam dryer at 80°C and kept there for 2 days. When dry, the distance between marks was measured by means of a Vernier calipers and the per cent linear shrinkage calculated as:

$$\frac{\text{Wet length--Dry length}}{\text{Dry length}} \times 100$$

Burning shrinkage--The briquettes were then burned in a gas-fired kiln, according to time temperature curve shown in Fig. IV. Three briquettes of each clay were drawn at each of the following temperatures:

Cone	Temperature
05	1050
1	1150
3	1190
5	1230
7	1270
9	1310
11	1350
13	1390

[illegible]

[illegible]

[illegible]

[illegible]	[illegible]
[illegible]	[illegible]

The burning shrinkage was determined by again measuring the distance between marks and calculated as:

$$\frac{\text{Dry length--Burned length}}{\text{Dry length}} \times 100$$

Porosity--The burned trials were weighed, immersed in water and heated for 2 hours under vacuum in a steel cylinder. The saturated and suspended weights were then determined and the percent porosity calculated as:

$$\frac{\text{Saturated weight--dry weight}}{\text{Saturated weight--Suspended weight}} \times 100$$

Softening point--Small cones of standard size were set in pots of alundum and kaolin mixture, with standard cones 26, 28, 30, 31 in the following arrangement:

△	△	△	△	△	△	△
26	clay	28	clay	30	clay	31

The pots were heated in an oil-fired Monarch furnace, using crude oil and low pressure air. The heat was raised at a rapid rate, so that the final temperature was reached in from 2 to 3 hours.

Load tests--The load tests adopted by Bleininger and Brown[4] was used for these tests. The clays were ground to pass 20 mesh, mixed with calcined Missouri flint fire clay, ground to pass $\frac{1}{4}$ mesh, and the whole pugged by hand. Standard size bricks were made by pressing this mixture which was a little stiffer than that used

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[4] Tech. Paper No. 7, U. S. Bureau of Standards, p. 46.

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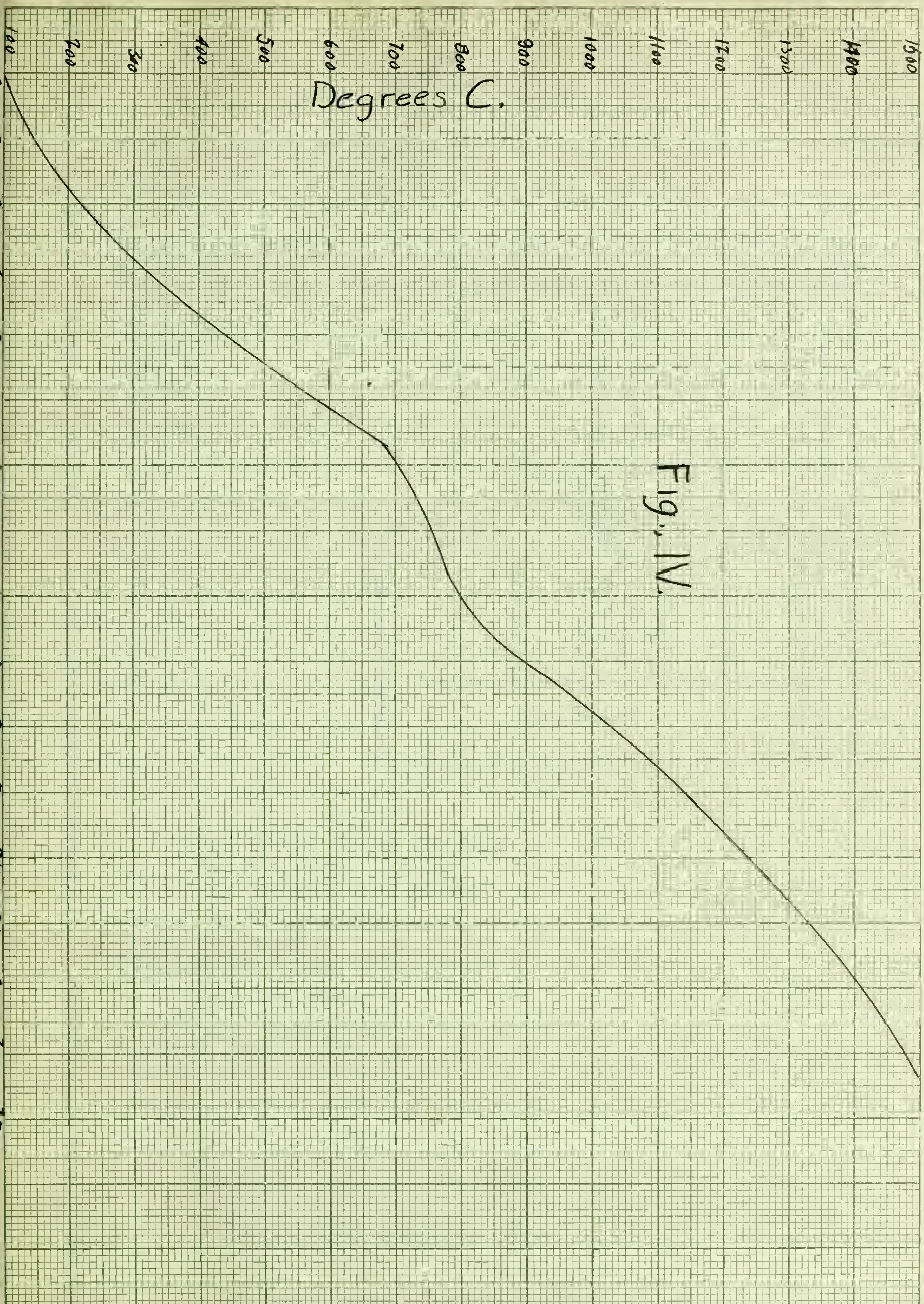
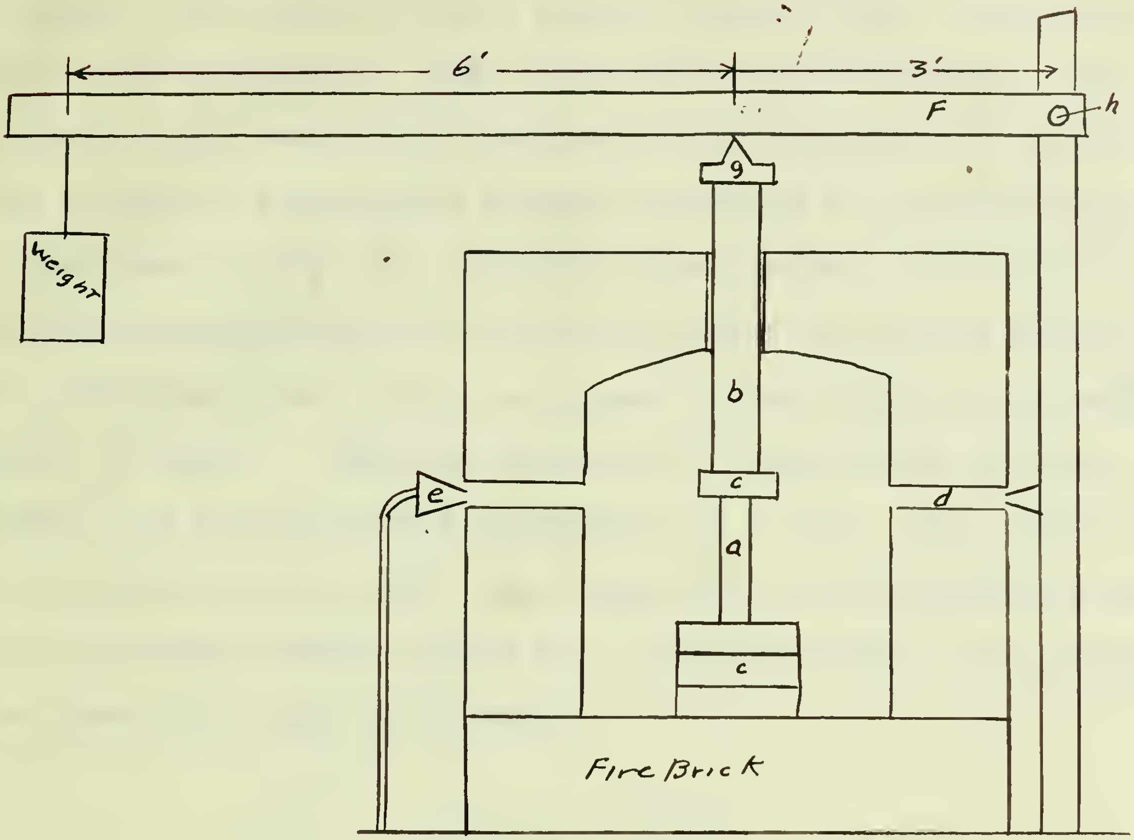

Degrees C.
100
200
300
400
500
600
700
800
900
1000
1100
1200
1300
1400
1500

Fig. IV.

Fig V.



a- Brick to be tested
b- Fire Clay column
c- Bearing Fire bricks
d- Flame inlet
e- Burner
F- I beam
g- Pin bearing

in making soft mud bricks in a hand power double acting repress machine. The amount of each material used in the mixture was 40% of the plastic and 60% of the calcined flint clays. The molded bricks were then dried for 2 days at 80°C in a steam dryer and burned in a gas-fired furnace according to the time temperature curve shown in Fig. IV. The brick was placed in an oil-fired furnace burning crude oil and high pressure air and a weight of 50 pounds per square inch was placed on the brick in the manner shown in Fig. V. The test consisted in heating the brick to 1350°C and holding this temperature for 1 hour. The rate of heating is shown in Fig. VI. The temperature was indicated by means of a platinum rhodium couple and a millivoltmeter both of which had been previously calibrated.

Results of Tests.

The results of these tests are shown in table I. Curves showing the burning, drying and total shrinkage are plotted against temperature in Fig. VII. The porosity temperature curves are shown in Fig. VIII.

The drying shrinkage of these clays is by no means excessive, for good plastic bond clays; also the burning shrinkage is much below the point which would cause difficulty in manufacture. The lowest and most uniform rate of shrinkage is shown by clay No. 3. This is entirely in accordance with its general flint-like structure. From the general appearance of the clays, it would be ex-

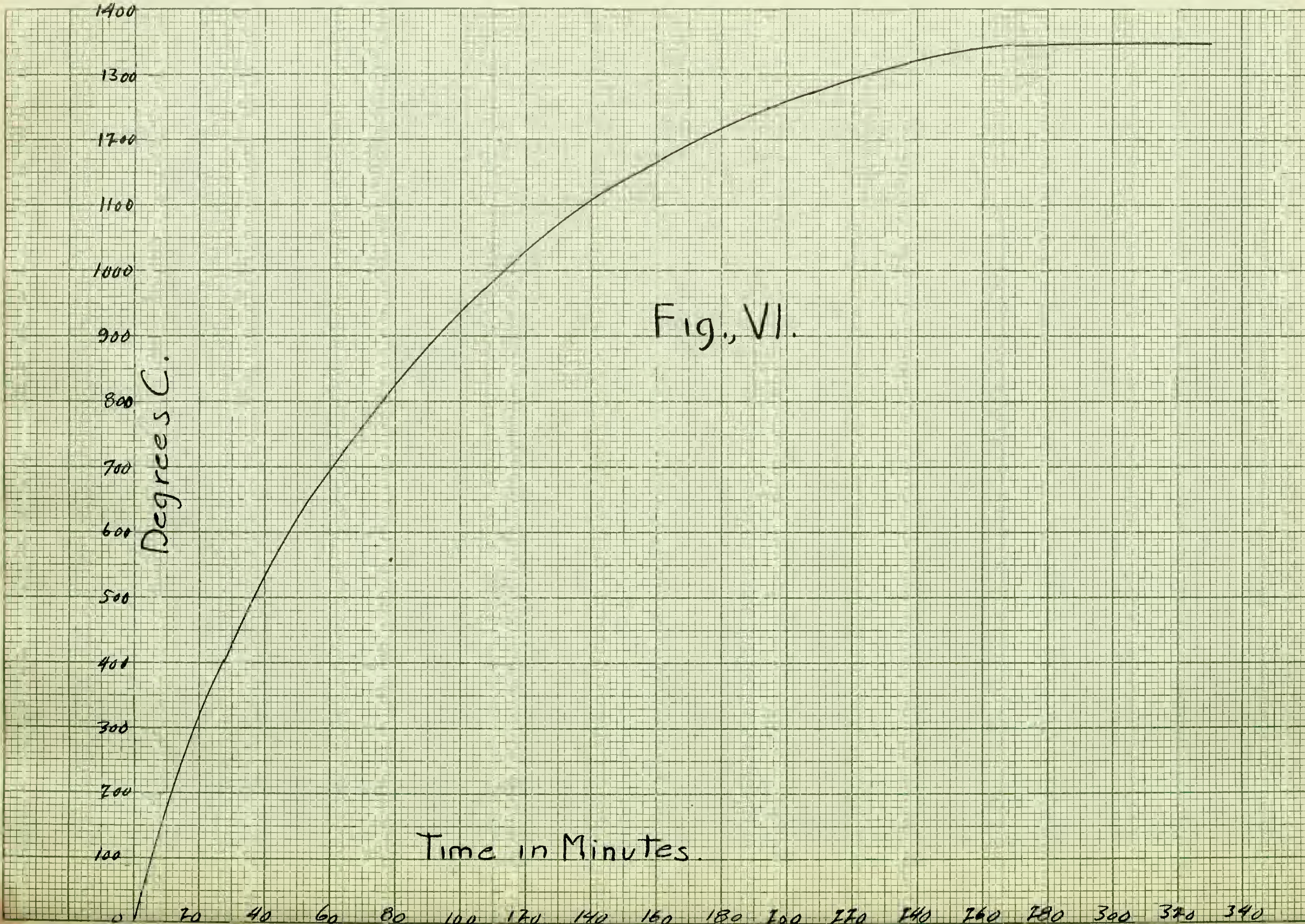

Degrees C.
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
20
40
60
80
100
120
140
160
180
200
220
240
260
280
300
320
340
Time in Minutes.

Fig., VI.

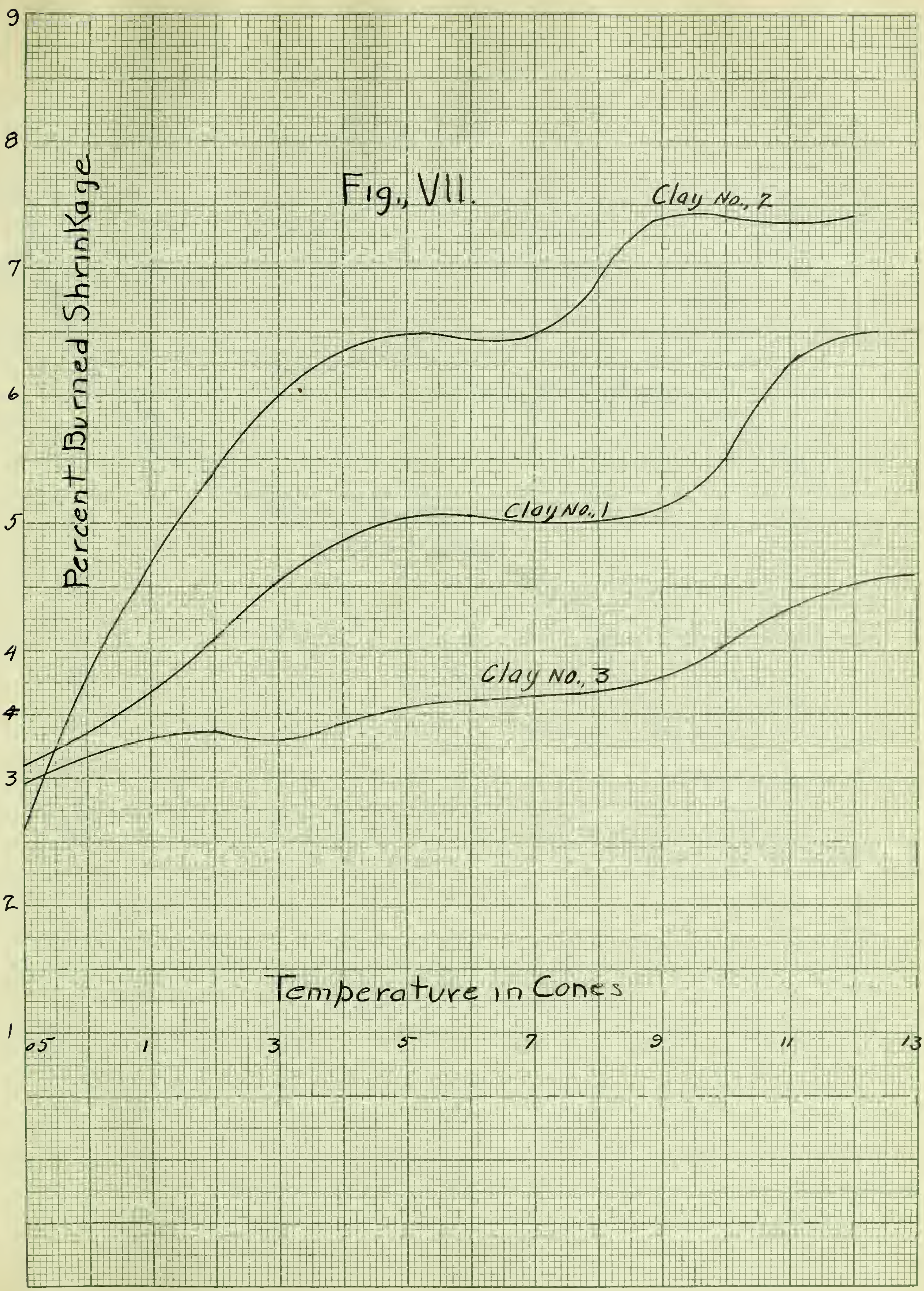


Fig., VII.
Percent Burned Shrinkage
Temperature in Cones
Clay No., 2
Clay No., 1
Clay No., 3
9
8
7
6
5
4
3
2
1
05
1
3
5
7
9
11
13

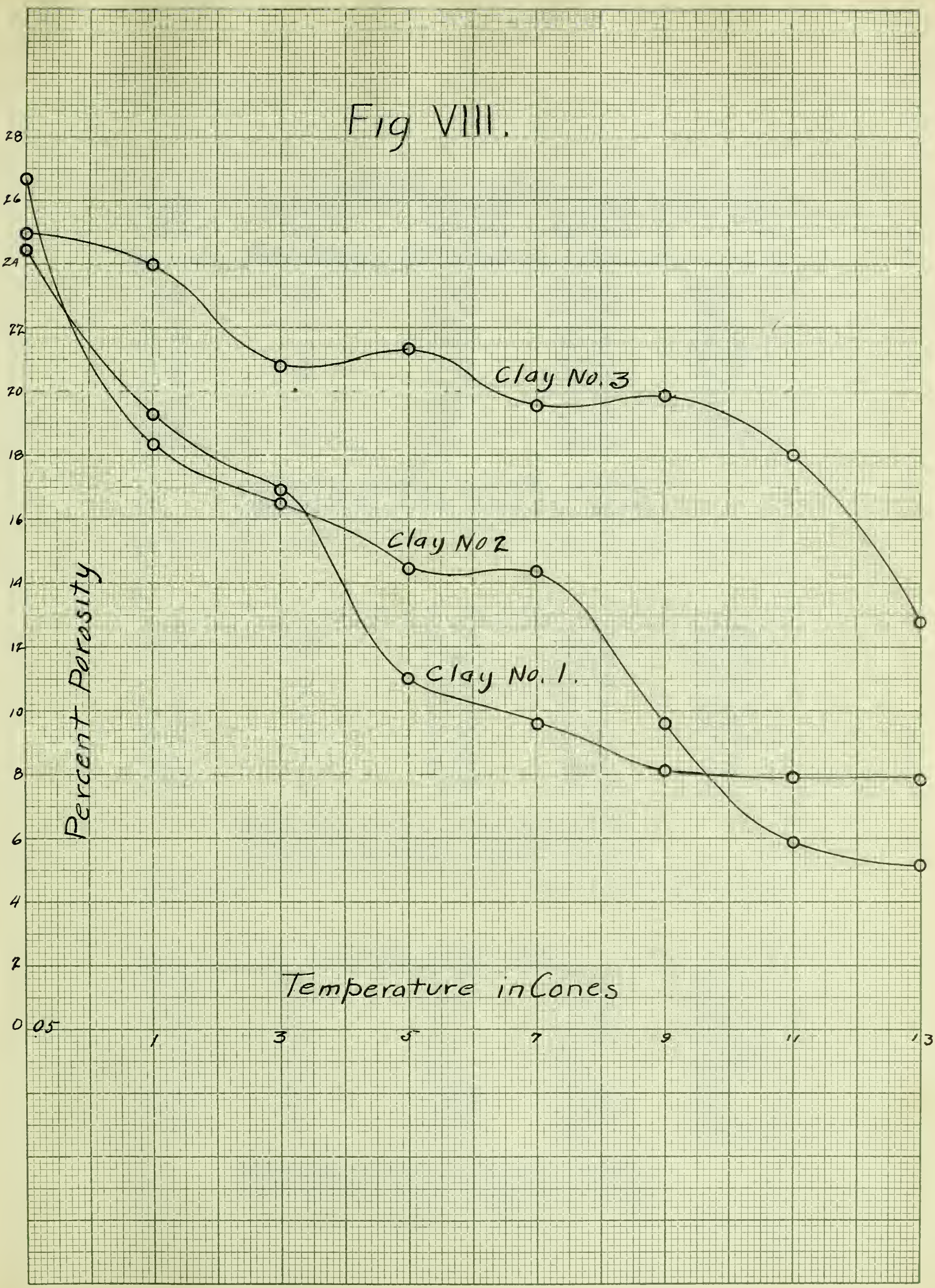


Fig VIII.
Clay No. 3
Clay No 2
Clay No. 1.
Percent Porosity
Temperature in Cones
0
2
4
6
8
10
12
14
16
18
20
22
24
26
28
05
1
3
5
7
9
11
13

--CLAY No. I--

Cone No.	Average Dry Shrinkage	Average Burning Shrinkage	Total Shrinkage	Porosity
05	8.49	3.06	11.55	24.6
1	"	3.68	12.17	19.7
3	"	4.55	13.04	16.9
5	"	5.02	13.51	10.92
7	"	5.00	13.49	9.8
9	"	5.07	13.56	8.
11	"	6.3	14.79	7.9
13	"	6.5	14.99	7.26

Softening point--cone 27, 1670°C.
Deformation under load--1 inch.

--CLAY No. II--

Cone No.	Average Dry Shrinkage	Average Burning Shrinkage	Total Shrinkage	Porosity
05	8.45	2.56	11.01	26.62
1	"	4.64	13.09	18.34
3	"	6.03	14.48	16.64
5	"	6.47	14.92	14.61
7	"	6.45	14.90	14.3
9	"	7.42	15.87	9.47
11	"	7.35	15.80	5.8
13	"	7.47	15.92	5.

Softening point--cone 30,1730°C
Deformation under load--7/8 inch.

--CLAY No. III--

Cone No.	Average Dry Shrinkage	Average Burning Shrinkage	Total Shrinkage	Porosity
05	8.4	2.95	11.35	24.9
1	"	3.3	11.75	24.
3	"	3.3	11.75	20.75
5	"	3.5	11.95	21.45
7	"	3.6	12.	19.71
9	"	3.85	12.25	19.9
11	"	4.32	12.72	18.
13	"	4.57	12.97	12.94

Softening point--cone 30, 1730°C,
Deformation under load--3/4 inch.

pected that No. 1 would show a greater shrinkage than No. 2, but the results show the opposite to be the case. The difference may be due to a larger amount of a different character of colloidal matter contained in No. 2.

The decrease in porosity as shown by the curves, is fairly regular for all three clays, and indicates an open structure of the molded pieces up to cone 13 (limit of test). Clay No. 3 comes well within Purdy's limits (Fig. I) for a No. 1 fire-clay; clay No. 2 at cone 11 falls just below the lower limit for a No. 2 fire-clay, being well above the limit at all previous temperatures, While clay No. 1 falls well within the limits for a No. 2 fire-clay.

The softening point determination shows samples Nos. 2 and 3 to be the most refractory. They may be classed as good bond clays, while No. 1, which softened at cone 27, is almost outside of the limits. The presence of small particles of pyrites, previously noted, probably accounts for the lower softening temperature.

The load tests showed clay No. 1 to be just inside the limits set by Bleininger and Brown (page 3); No. 2 shortening 7/8 inches, may according to this test, be called a No. 1 fire-clay, while No. 3, with a deformation of only $\frac{3}{4}$ inches, is well within the specified limits.

General Conclusions.

The results indicated by the tests show that the clays studied meet the requirements for good refractory bond clays, although they do not entirely fulfill the qualifications which have been previously mentioned.

Clay No. 1 has the required low drying and burning shrinkage and the character of its porosity curve places it in the class of clay No. 2 fire-clays as given by Purdy. In its behavior under load conditions, it ranks in class No. 1 (Bleininger and Brown). The low softening point, cone 27 however, detracts from its value. Clay No. 2 has the required low shrinkage. The porosity curve falls within Purdy's limits for a No. 2 fire-clay. Its behavior under load conditions places it in class No. 1 and its relatively high softening point, (cone 30) is above that required for a No. 2 fire-clay. Clay No. 3 has a very low and regular shrinkage. The porosity curve lies in the field of No. 1 fire-clays. The load test shows it to be well within specifications for a No. 1 fire-clay, and its softening temperature of cone 30 is just below that required for a No. 1 fire-clay.

Taking all the test results into consideration, it would be seen that clays Nos. 1 and 2 should be classed as No. 2 refractory fire-clays, while No. 3 should rank as a No. 1 bond clay.